Standard Procedure for Grain Size Analysis

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Note: The effective date is considered to be the last approval date.

Revision Page

Date	Rev #	Summary of Changes	Sections
	1	Initial Approval	
2/14/03	2	modification of method to include separation of wet and dry components	all
4/10/03	3	Details of New Laboratory	title page
2/24/10	4	Convert to Word, format, signatures	all
3/17/15	5	Add input of data into locked spreadsheets as well as grain size logbook	6,7

Table of Contents

1.0 Purpose of Method

Sediments are made up of varying particle sizes. This test is designed to determine the grain size fractions that make up a sediment sample. Fine grained sediments having diameters of 100 microns or fewer exhibits a high degree of cohesiveness and a large surface area. This particular physico-chemical characteristic has been shown to affect the bioavailability of toxic agents and identifying its presence can improve the weight of evidence required for interpreting a health assessment of organisms indigenous to a particular area.

2.0 Summary of Method

An aliquot of sediment is split into a sand fraction and a mud fraction. The sand fraction is broken down into its components using a dry sieve method. The mud fraction is determined via a pipette technique. A final calculation is performed on all dried components, both sand and mud fractions, to determine the grain size breakdown of the sediment sample(s) under study.

3.0 Health and Safety

3.1 If samples are from an area of known toxicity or if the samples are sulfurous or noxious in nature, then all work should be done using a wet sieving technique.

3.2 All personnel should follow standard safety procedures that include wearing a laboratory coat, safety glasses with splash guards and gloves.

3.3 Efforts are made during planning to ensure that dry sand component breakdown is actually necessary according to DQOs.

3.4 In instances where a breakdown of the dry sand component is needed all dry sieving takes place in the Rm 205 sieve shaker hood specifically designed with a HEPA filter for this activity.

4.0 Equipment and Materials

- Dried and pre-weighed glass beakers, 100 and 500ml
- Drying oven
- Weight calibration checked top loading balance
- Spatulas
- Graduated cylinders, 1 and 2L
- Disposable pipettes, 25 ml
- Stop watch

- Sieve set (#4, #10,#40,#200)
- Sieve shaker
- Rm 205 Containment Hood
- Buckets, white, 5 -gallon
- Desiccator
- Grain Size logbook
- Oven logbook
- Locked Excel Spreadsheet (ECA S drive)

5.0 Reagents

- Deionized Water
- Sodium hexametaphosphate solution (6.2g/L)

6.0 Procedure

6.1 Sample Handling

6.1.1 Sediment samples are to be de-watered in the field by decantation and then homogenized at the sampling location prior to being allocated into sample containers.

6.1.2 Sediment samples for grain size analysis are to be placed in 12" x 12" zip-loc bags filling to half volume. Sediment samples can be transported and stored at room temperature.

6.2 Sample Preparation

6.2.1 Take the sample as delivered from storage and allow to equilibrate to room temperature, if necessary.

6.2.2 With the sample still in the bag, mix and "work" the sample to uniform consistency making sure that any aggregates are broken up.

6.2.3 Using a top-loading balance which was calibration checked the day of use, weigh out approximately 100 grams of sediment into a clean 1L beaker.

6.2.4 Add 13 ml of sodium hexametaphosphate (6.2 g/L) to the beaker with a disposable pipette. Add 77 ml of deionized water to the same beaker.

6.2.5 Gently mix on a stir plate for 30 minutes.

6.3 Separation of Sand and Silt Component (EPA Region II Method Reference)

Separate the sand component (>75u) from the mud (<75u) by wet sieving, with a clean # 200 sieve.

Note: Once separated, the sand and mud fractions can be worked on simultaneously

6.3.1 Place the sieve above a clean 5 gallon bucket which has a 1 liter and 2 liter mark on the inside.

6.3.2 Pour the sediment slurry onto the #200 sieve.

6.3.3 With deionized water, rinse the slurry through sieve until the rinse water runs clear, **paying attention that the final volume in the bucket is ≤ 1 liter.**

6.4 Sand component - retained on the #200 Sieve (ASTM Reference)

6.4.1 Dry and pre-weighed 500 ml glass beaker. Record weight in grain size analysis logbook and Excel Spreadsheet template. With a minimal amount of DI, transfer the sample retained on the # 200 sieve into the 500 ml glass beaker to a dry.

6.4.2 Turn on the oven in advance and set to 110°C and record this in the oven logbook. Dry the beaker with sand in a 110°C oven for four hours. Also note in the grain size logbook which oven and temperature "in" with initials and date.

6.4.3 If left overnight in the oven or desiccator be sure to perform the daily balance calibration check before proceeding. Remove the beaker with sand from the oven and allow cooling to room temperature in a desiccator then weigh the dried beaker with sand to the nearest 0.1 grams. Record this "sand + beaker" weight in the grain size analysis logbook.

6.4.4 Place the beaker with sand back in the oven for an additional hour noting in the grain size logbook which oven and temperature "in" with initials and date. Take at least two measurements one hour apart to ensure an accurate final beaker with sand dry weight (i.e. <0 .1% change). Record this final weight in the grain size analysis log book and Excel Spreadsheet template.

6.4.5 Subtract the weight of the beaker from step 6.4.1 from the weight determined in step 6.4.4 and record as the dry weight of the sand component. *This is automatically calculated in the Excel spreadsheet.*

6.4.6 Clean, dry and pre-weigh, a set of sieves (normally 4, 10, 40, 140 and pan). Record these

weights in the grain size analysis log book and Excel Spreadsheet template.

6.4.7 Set the sieve stack up, coarsest on top to finest with a pan at the bottom.

6.4.8 Place the stack in the mechanical shaker inside the Rm 205 sieve hood and turn on the hood using toggle on left side of unit. Carefully and completely, pour the dried sand component from step 6.4.4 onto the top of the stack. Place a sieve stack cover over the top sieve, lock the stack down, set the shaker to run on step 4 for 15 minutes and turn it on.

6.4.9 Carefully disassemble the stack and weigh each sieve with remaining material and then pan on the top loading balance, recording the sieve weights in the grain size analysis log book.

6.4.10 Reassemble the stack, not including sieves with no material remaining on them, and repeat steps 6.4.7, 8 and 9. Repeat until a constant weight of < 4% of the previous reading (based on only the sample weight not sample + sieve) or 0.1 grams, whichever is greater is reached. Record these final weights in the grain size analysis log book and Excel Spreadsheet template.

6.5 Mud Component - passed through #200 Sieve

6.5.1 Take the portion of the sample in the white bucket and rinse into a 1 L graduated cylinder.

6.5.2 Bring the volume in the cylinder up to 1 L with deionized water and cover the top tightly with para-film.

6.5.3 If in an unusual instance more than 1 L was necessary to rinse, completely transfer the contents in the 1L cylinder into a 2L graduated cylinder, bring the final volume to 2L and cover the top tightly with para-film.

6.5.4 Record the final volume in the grain size analysis log book.

6.5.5 Allow sample to come to room temperature.

6.5.6 Thoroughly mix the sample in the cylinder by inverting the cylinder several times. **Immediately** after mixing, begin timing with a stop watch. At **exactly the 20 second mark, out of the cylinder, remove 20.0 mls of sample from a water depth of exactly 20 cm** with a disposable 25 ml pipette (for ease the 20 cm mark can be pre-scribed with a "sharpie" onto the cylinder) and place in a, pre-weighed and recorded in the grain size analysis log book and Excel Spreadsheet template, 100 ml beaker, rinsing the outside of the pipette into the beaker as well.

6.5.7 Turn on or adjust the oven in advance and set to 60°C. Record this in the oven logbook. Dry the 100 ml beaker with the 20 mls of sample at 60°C overnight. Note in the grain size logbook which oven and temperature "in" with initials and date.

6.5.8 Remove the "dry mud sample + beaker" and allow to cool to room temperature in a desiccator.

6.5.9 On a calibration checked, top loading balance weigh the "dry mud sample + beaker" and record in the grain size analysis log book and Excel Spreadsheet template.

6.5.10 Subtract the weight of the dry 100 ml beaker from the dried "mud" weight determined in step 6.5.9 and record in the grain size analysis log book. *This is automatically calculated in the Excel spreadsheet.*

7.0 Calculations (*See locked Excel spreadsheet in the S drive under ECA/EMT/Biology Lab/laboratory forms/locked spreadsheets)*

7.1 When using a dispersant of sodium hexametaphosphate at a concentration of 6.2g/L, subtract 0.00087g from the weight determined in 6.5.10. *This is automatically performed in the Excel spreadsheet.*

7.2 To get the total dry weight of mud take the dry mud weight obtained in step 7.1, and multiple by dilution factor. *This is automatically calculated in the Excel spreadsheet.* For example, if the final volume in step 6.5.4 was 2L and you removed 20 mls the dilution factor is 100. ***Note the actual dilution factor must be entered into the spreadsheet calculation function.***

7.3 Record this weight in the grain size analysis log book as the total dry weight of the silt/clay component i.e. <75u. *This is automatically calculated in the Excel spreadsheet.*

7.4 Add the weight value from step 7.3 to the weight determined in 6.4.5 and record this weight in the grain size analysis log book as the total dry weight on the sample. *This is automatically calculated in the Excel spreadsheet.*

7.5 Calculate the weight of each sand size component by subtracting the weight of the sieve from the weight of the sand plus sieve determined in step 6.4.10. *This is automatically calculated in the Excel spreadsheet.*

7.6 Calculate the dry % of total for each size component, both sand and silt/clay, by dividing the individual component dry weight obtained in 6.4.10 by the total weight from step 6.4.5 and multiply by 100. *This is automatically calculated in the Excel spreadsheet.*

7.7 Report all final results as the % fraction in dry weight.

8.0 Quality Control

8.1 The balance used for weighing is checked for daily calibration and the check is recorded in the calibration log book.

8.2 Oven temperatures are recorded in the oven logbook.

8.3 All sample numbers, weights and analyst are documented in a hard bound analysis log book.

8.4 Under the site specific QAPP a duplicate grain size analysis may be performed to identify the precision of the sampling and analysis procedure.

8.5 All sieves used for the analysis conform to ASTM standard sieve size specification.

9.0 Waste Management and Pollution Control

9.1 Sediment from this procedure is placed in site specific labeled containers marked as "Sediment waiting for analyses" prior to disposal by the H&S officer.

9.2 If the sediments are known to be contaminated, any rinse water used in the process is stored in Rm205, in a site specific labeled container(s) as "site ID and awaiting analyses for disposal", with date, project name and initials. The lab lead is made known of the analysis material to be disposed of.

10.0 Records Management

10.1 All records of sample manipulation and weighing is recorded in the grain size analysis logbook.

10.2 All weights are also recorded for results calculation in a protected Excel spreadsheet.

10.3 Electronic report files are maintained in the ECA S drive under biology laboratory and grain size.

10.4 Hard copy reports are maintained in a project folder with other pertinent information in the ECA Biology Laboratory file in Rm 152.

11.0 References:

- ASTM method D422-63

- EPA Region II Grain Size Method